^{14}C MEASUREMENTS OF ICE SAMPLES FROM THE JUVFONNE ICE TUNNEL, JOTUNHEIMEN, SOUTHERN NORWAY—VALIDATION OF A ^{14}C DATING TECHNIQUE FOR GLACIER ICE

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ABSTRACT. Establishing precise age-depth relationships of high-alpine ice cores is essential in order to deduce conclusive paleoclimatic information from these archives. Radiocarbon dating of carbonaceous aerosol particles incorporated in such glaciers is a promising tool to gain absolute ages, especially from the deepest parts where conventional methods are commonly inapplicable. In this study, we present a new validation for a published ^{14}C dating method for ice cores. Previously ^{14}C-dated horizons of organic material from the Juvfonne ice patch in central southern Norway (61.676°N, 8.354°E) were used as reference dates for adjacent ice layers, which were ^{14}C dated based on their particulate organic carbon (POC) fraction. Multiple measurements were carried out on 3 sampling locations within the ice patch featuring modern to multimillennial ice. The ages obtained from the analyzed samples were in agreement with the given age estimates. In addition to previous validation work, this independent verification gives further confidence that the investigated method provides the actual age of the ice.

INTRODUCTION

Glaciers and ice sheets comprise valuable information about past climatic and environmental conditions on Earth. Precise age-depth information of any such archive is of fundamental importance in order to retrieve legitimate conclusions from paleoclimate records. In studies of ice cores from high-alpine glaciers, however, dating is a non-trivial task mainly due to complex influences such as glacier flow, accumulation, ablation, and layer thinning resulting in a strongly non-linear age-depth relation (Knüsel et al. 2003). Particularly in the deepest parts of a glacier, where thinning of annual layers does not allow for conventional annual layer counting (ALC) on seasonally varying parameters (Thompson et al. 1990) and where the application of ice flow models is often hampered by the complex small-scale topography of bedrock (Lüthi and Funk 2001), other techniques are required to establish a precise age-depth relation. Radiocarbon dating has successfully been applied to date natural ice samples in cases where sufficient carbon containing material such as plant fragments, insects, or other organic remains were incorporated (Thompson et al. 1998, 2006; Nesje et al. 2011). We developed a ^{14}C method for dating ice-core samples utilizing the ubiquitous trace amounts of carbonaceous aerosol particles embedded in the ice matrix and applied it to natural ice samples from various glaciated regions all over the globe including some in-depth investigations of basal ice-core sections (Jenk et al. 2009; Kellerhals et al. 2010; Wientjes et al. 2012; Herren et al. 2013).

Here, we present further validation measurements of the ^{14}C method for ice cores as introduced by Sigl et al. (2009). In multiple measurements, micro-amounts of particulate organic carbon (POC) were extracted from clear ice samples from a Norwegian ice patch and were ^{14}C dated with our method. A previous study used organic macro-subfossils contained within dark ice layers in the same ice patch for conventional ^{14}C dating (Nesje et al. 2011). These ages were then used as reference dates for the results obtained from the POC fraction (this study). This investigation was a

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unique chance since we could for the first time compare our method with the ages of samples containing sufficient organic material for routine ^{14}C dating, which is a rare case for ice cores.

METHOD

The method applied and discussed in this study was initially developed as a tool for source apportionment of ambient carbonaceous aerosols (Szidat et al. 2004) and later adapted to the ^{14}C analysis of carbonaceous particles in glacier ice (Jenk et al. 2006). The basic principle of the method as a dating tool for preindustrial ice is schematically shown in Figure 1.

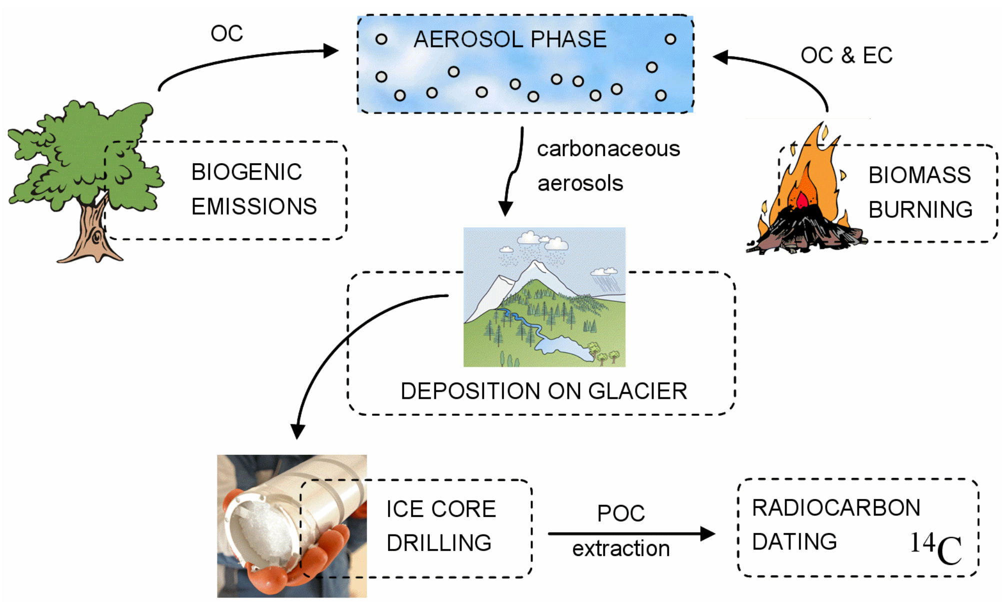

Figure 1 Schematic of the basic principle underlying the ^{14}C method illustrating preindustrial sources (no fossil contribution) and pathways of carbonaceous aerosols in an ice core: OC (organic carbon, hydrocarbons of low to medium molecular weight); EC (elemental carbon, highly polymerized hydrocarbons); POC (particulate organic carbon, water-insoluble OC).

A more detailed description of the method including necessary corrections was presented by Jenk et al. (2007). Briefly, the ice samples are decontaminated by removing the outer layers in a 3-step process (cutting with a band saw, scraping with a scalpel, rinsing with ultrapure water) to remove potential contamination from sampling and handling operations. Melted samples are then filtered through quartz fiber filters (Pallflex Tissuquartz, 2500QAO-UP; prebaked) and carbonates are removed by acidification of the filter residue with 0.2M HCl. Dried filters are combusted in a 2-step process where the fractions of POC and elemental carbon (EC) are separated (10 min at 340 °C and 12 min at 650 °C, respectively) followed by cryogenic trapping and manometric quantification of the evolving CO_2. Finally, the CO_2 samples are sealed in glass ampoules that are introduced to the gas handling system of the 200kV accelerator mass spectrometer system MICADAS for ^{14}C determination at the ETH Laboratory of Ion Beam Physics (Ruff et al. 2007; Synal et al. 2007). EC fractions were isolated for each sample but not used for dating due to the rather low concentrations. Procedural blank estimation was carried out during the sample series by using blank ice (frozen ultrapure water, 18 MΩ cm) subjected to the identical analytical procedure as applied for the real samples. Usually, 3 filters were pooled for analysis to obtain sufficient material for quantification and subsequent

AMS measurement (>3 µg C). During the period of sample analysis for this study, a procedural blank input of 1.4 ± 0.3 µg POC ($m_{C,proc.blank}$) with a fraction of modern ($Fm_{proc.blank}$) of 1.2 ± 0.4 per filter was determined and corrected for according to:

$$Fm_{corr} = \frac{m_{C,\,sample} \cdot Fm_{sample} - m_{C,\,procblank} \cdot Fm_{procblank}}{m_{C,\,sample} - m_{C,\,procblank}}$$

where $m_{C,sample}$ represents the measured carbon mass in the respective sample and Fm_{sample} denotes the measured fraction of modern of the sample. A total of 11 ice samples (0.190–0.368 kg) representing the 3 sampling locations (see below) were dated using the POC fraction.

PREVIOUS VALIDATION WORK

Previous validation work on the dating method was carried out by comparing the ^{14}C ages with results from independent dating techniques applied on the same ice-core sections (Jenk et al. 2009; Sigl et al. 2009). Approaches included i) the measurement of old, well-dated ice from Greenland, GRIP, 72.58°N, 37.64°W (Vinther et al. 2006; Jenk et al. 2009); ii) matching ALC-derived ages from alpine ice cores and ^{14}C; iii) analyzing ice surrounding an identified volcanic eruption in an ice core from Illimani (16.62°S, 67.77°W); and iv) comparing the ^{14}C-dated Pleistocene/Holocene transition in the δ^{18}O record from the Illimani core with δ^{18}O trends from other ice cores containing this feature. Greenland samples mostly contained very low carbon amounts of <5 µg kg^{-1} and did not result in meaningful dating (Jenk et al. 2009). ALC-derived timescales from high-alpine ice cores usually reach back just a few centuries, which makes it difficult to compare them with ^{14}C dates as these perform less reliable for such young ages (due to fossil carbon bias and flattening of the ^{14}C calibration curve). A tentative ALC chronology for an ice core from Mercedario (31.97°S, 70.12°W), however, was confirmed by 2 ^{14}C dates within their 1σ range (Sigl et al. 2009). Four samples surrounding the AD 1258 volcanic eruption in the Illimani core resulted in very consistent ages from 800 to 1120 cal yr BP (1σ range), thus overestimating the expected age by roughly 200 yr, an acceptable dating accuracy on the Holocene timescale (Sigl et al. 2009). The δ^{18}O record from Illimani, based on a ^{14}C-derived timescale, showed a strong similarity with δ^{18}O records from other Andean ice cores (Huascaran, 9.11°S, 77.61°W; Sajama, 18.10°S, 68.88°W) during the Pleistocene/Holocene transition phase around 8–12 kyr BP (Thompson et al. 1995, 1998; Sigl et al. 2009). Considering these validation efforts, the method seemed to yield reasonable dates representing the age of the ice, yet very much dependent on the amount of available carbon. A detailed description of the validation work summarized above was published by Sigl et al. (2009). The validation attempt aimed for in this study focuses for the first time on the comparison of our ^{14}C dates with their expected ages as deduced from conventionally ^{14}C dated organic rich layers in an ice patch.

STUDY SITE AND SAMPLING

Ice samples investigated in this work were collected in 2010 from the Juvfonne ice patch. Juvfonne is a small perennial ice patch in the Jotunheimen Mountains in central southern Norway (61.676°N, 8.354°E, Figure 2) currently covering 0.2 km^2 at a mean altitude of 1925 m asl. Ground penetrating radar (GPR) soundings revealed a maximum ice thickness of 17–19 m (Oedegaard et al. 2011).

Increasing interest in those ice patches arose as a result of the extreme melting that the region experienced in 2006 when numerous historical artifacts had been released by the retreating ice (Nesje et al. 2011; Oedegaard et al. 2011). In May 2010, an approximately 30-m-long ice tunnel was excavated in the Juvfonne ice patch. Stratigraphic examination of the ice patch, both with GPR and from observations within the ice tunnel, revealed consistent findings: While the upper horizons are virtu-

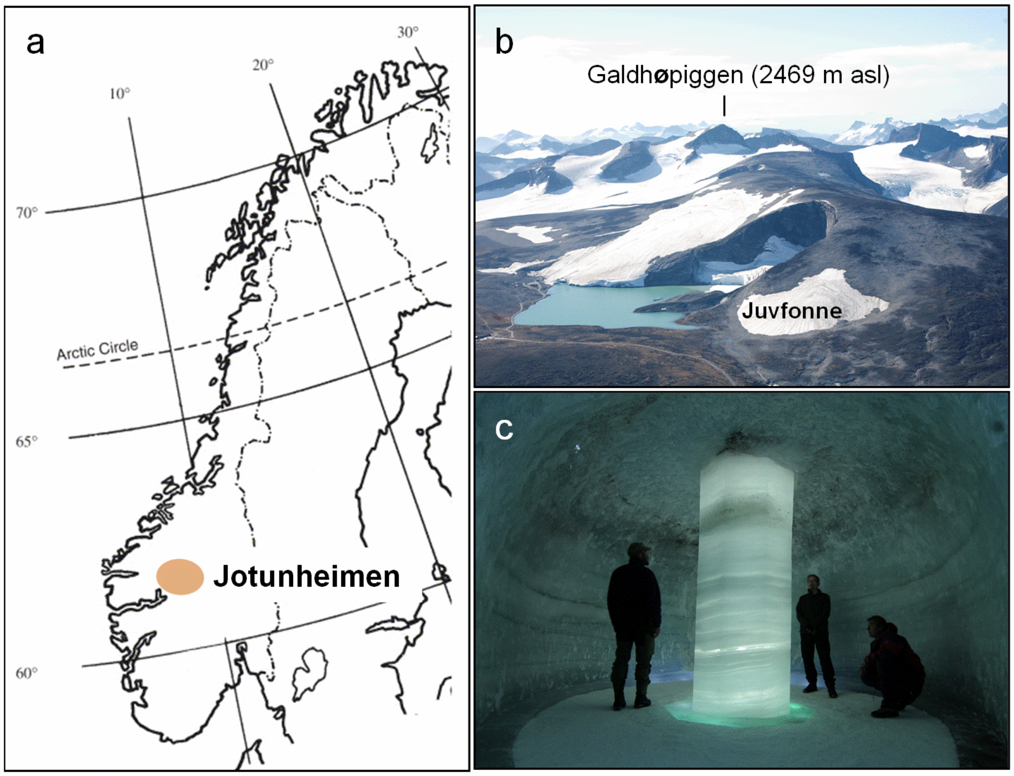

Figure 2 Study site impressions. (a) Location of the study area (brown dot) in southern Norway. (b) Aerial photograph of the Juvfonne ice patch and its proximity in the Jotunheimen mountains. Photograph by Helge J Standal. (c) Inside the investigated ice tunnel. Note the layering of the ice including some dark horizons. Photograph provided by KlimaPark2469.

ally parallel to the present surface, the deeper ice layers are slightly curved and describe a distinct angular unconformity with the surface layers, probably caused by deformation when the ice patch grew during the Little Ice Age or earlier (Nesje et al. 2011). Inside the ice tunnel, several up to 5-cm-thick dark layers were detected comprising minerogenic as well as fine-detritus organic residues, which were interpreted as previous ice-patch surfaces (Nesje et al. 2011). In an earlier study, samples were extracted from the organic-rich layers to identify when the ice patch formed. Conventional ^{14}C measurements on those samples (Table 1) indicated that the deeper part of the ice patch might be as old as 3200 yr cal BP (Nesje et al. 2011). For the present study, 2 different locations (JUV 1 and JUV 2) adjacent to the previously dated organic-rich layers within the ice tunnel were sampled. Blocks of ice (~15 × 15 × 40 cm) were extracted with a chainsaw and were subsequently divided into smaller pieces prior to analysis. The sampled spots were characterized by clear ice suitable for our dating method based on carbonaceous aerosol particles. The expected age ranges of the samples were approximated based on the ages of the independently ^{14}C-dated organic layers and their location with respect to the samples to be tested. One additional sample (JUV 3) was collected from young ice at the margin of the ice patch. An overview of the discussed samples together with their expected ages is given in Table 1.

Table 1 Summary of the ^{14}C-dated Juvfonne samples. The identified outlier JUV 1_1/2 is given in parentheses. Means of the respective sample series are given in bold. Italic data represent the reference ages used to constrain the expected age ranges (see text; data from Nesje et al. 2011). Calibrated using OxCal v 4.1 (Bronk Ramsey 2009) and the IntCal09 data of Reimer et al. (2009). Data are given with 1σ uncertainties.

Sample ID	AMS Lab nr	Ice (kg)	POC (μg)	*Fm*	^{14}C age (yr BP)	Age (cal yr BP)
(JUV 1_1/2)	(ETH 43442.1.1)	(0.368)	(24.3)	(0.617 ± 0.026)	(3880 ± 340)	(3890–4820)
JUV 1_3	ETH 43555.1.1	0.282	20.2	0.766 ± 0.029	2144 ± 300	1740–2680
JUV 1_4	ETH 43557.1.1	0.216	9.2	0.719 ± 0.064	2650 ± 710	1900–3680
JUV 1 mean	**—**	**—**	**—**	**0.742 ± 0.026**	**2390 ± 280**	**2070–2780**
Juv-1051	*Poz-36460*	—	—	*0.692 ± 0.003*	*2960 ± 30*	*3078–3207*
Juv-1053	*Poz-37878*	—	—	*0.826 ± 0.003*	*1535 ± 30*	*1378–1419*
JUV 2_1	ETH 43443.1.1	0.223	28.3	0.881 ± 0.023	1020 ± 210	740–1170
JUV 2_2	ETH 43445.1.1	0.190	10	0.792 ± 0.066	1870 ± 670	1180–2720
JUV 2_3	ETH 43559.1.1	0.272	16.5	0.870 ± 0.035	1120 ± 320	730–1320
JUV 2_4	ETH 45109.1.1	0.230	19.4	0.869 ± 0.031	1130 ± 280	780–1300
JUV 2 mean	**—**	**—**	**—**	**0.853 ± 0.016**	**1280 ± 150**	**1010–1320**
Juv-1054	*Poz-37879*	—	—	*0.838 ± 0.003*	*1420 ± 30*	*1300–1338*
JUV 3_1	ETH 42845.1.1	0.291	54.8	1.124 ± 0.0013	modern	modern
JUV 3_2	ETH 42847.1.1	0.267	43.1	1.094 ± 0.015	modern	modern
JUV 3_3	ETH 42849.1.1	0.326	46.8	1.155 ± 0.015	modern	modern
JUV 3_4	ETH 43446.1.1	0.212	44.4	1.164 ± 0.017	modern	modern
JUV 3 mean	**—**	**—**	**—**	**1.134 ± 0.007**	**modern**	**modern**

RESULTS AND DISCUSSION

Samples from 3 different locations were analyzed and repeated measurements were carried out for each site. Since sampling locations are distinct and independent from each other, the respective results will be discussed separately. A compilation of the investigated samples and the ^{14}C data used for the estimation of the expected ages is listed in Table 1 and illustrated in Figure 3. Dates are given in calibrated years before present (cal yr BP) with their 1σ uncertainty range, where not stated otherwise.

JUV 1

POC concentrations in this sample sequence varied from 43 up to 72 μg kg^{-1} (60 ± 15 μg kg^{-1}), a sufficient amount for reliable dating. The expected age for that site ranges from 1378 to 3207 cal yr BP as previously estimated based on the surrounding, conventionally ^{14}C-dated samples *Juv-1051* and *Juv-1053* as reported by Nesje et al. (2011). One sample (JUV 1_1/2) out of the JUV 1 series giving an age of 3890–4820 cal yr BP was considered an outlier. The outlier was identified due to its outstandingly high POC/EC ratio (14.7 compared to 4.5 ± 1.1 for the remaining samples), a value suggesting contamination during sample processing. Samples JUV 1_3 and JUV 1_4 resulted in 1740–2680 and 1900–3680 cal yr BP, respectively. The average of the JUV 1 measurements is 2070–2780 cal yr BP and lies perfectly within the expected period.

JUV 2

This sample series exhibited POC concentrations between 53 and 127 μg kg^{-1} (81 ± 33 μg kg^{-1}). The reference age for this sample was given by the date of adjacent layer *Juv-1054*, 1300–1338 cal yr BP (Nesje et al. 2011). Due to the distinct angular unconformities observed within the ice layers in the vicinity of JUV 2, the *Juv-1054* reference age was rather used as an indication for the expected age rather than a definite date. Three out of 4 subsamples for JUV 2 gave very consistent ages from 730 to 1320 cal yr BP, a range in line with the reference age. The fourth sample (JUV 2_2) gave a relatively older value of 1180–2720 cal yr BP with a considerably larger 1σ range. The available carbon amount was rather small (10 µg) leading to the larger uncertainty. No indications, however, were observed suggesting that JUV 2_2 would be a contaminated outlier. Given the complexity in the stratigraphy the JUV 2 average value of 1010–1320 cal yr BP agrees very well with the age of the neighboring reference sample within the uncertainties. To better resolve the stratigraphic age sequence in this part of the ice patch, more detailed information on the layering and further measurements are needed.

JUV 3

Sample JUV 3 from the frontal margin of the ice patch had by far the highest carbon content in this study. Carbonaceous particle concentrations for the 4 subsamples varied from 144 to 209 µg kg^{-1} (176 ± 29 µg kg^{-1}). The sampling site is supposed to represent modern ice, a fact that could unambiguously be confirmed by our measurements. Values for Fm ranged from 1.095 ± 0.015 to 1.164 ± 0.017 with an average of 1.134 ± 0.007. POC of this modern sample comprises 3 emission fractions of different ^{14}C signatures (Jenk et al. 2006): fresh biomass originating from the same year the ice layer formed, biomass burning integrating biomass of several decades before deposition and fossil-fuel combustion that does not contribute any ^{14}C (Fm = 0). This situation complicates the absolute dating of JUV 3. Nevertheless, the measured Fm values prove that this sample is younger than 53 yr before collection.

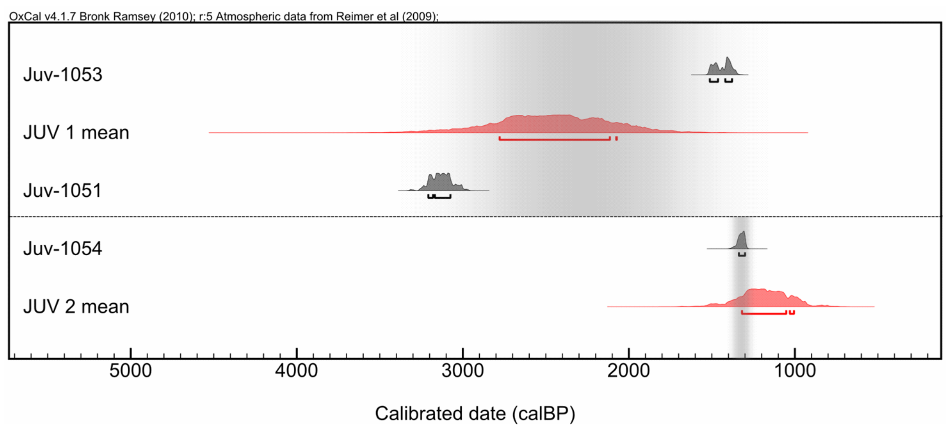

Figure 3 Calibrated mean ages of JUV 1 and JUV 2 (red distributions). Also shown are the calibrated ages from the conventionally ^{14}C-dated samples of organic layers (gray distributions: Juv-1054, Juv-1053, Juv-1051 from Nesje et al. 2011), used to obtain the expected age ranges indicated by the shaded areas. Brackets under the probability distributions represent the age range containing 69% probability for the true age.

CONCLUSIONS

A further line of evidence for the applicability of the ^{14}C method for ice-core dating was thus accomplished. Analyzed samples met the expected age ranges reasonably well within their uncertainties. While the range for JUV 1 was clearly confirmed, JUV 2 seemed to represent an ice layer slightly younger than the dated organic reference horizon, which is quite possible considering the complex layering around the sampling location. Modern ice at the margin of the ice patch was definitely confirmed by JUV 3, a fact not to be underestimated as it helps to refute concerns about potential systematic age offsets towards very much older ages (e.g. reservoir effects, pre-aged carbon). Along with previous validation efforts, the data obtained in this study provide additional confidence for our method to provide the true age of the ice.

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